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Space Science Laboratory

RE-ENTRY PHYSICS RESEARCH (U)
SEMI-ANNUAL TECHNICAL SUMMARY REPORT

Prepared by

GENERAL DYNAMICS/CONVAIR

San Diego, California 92112

December 1965

Prepared for

ADVANCED RESEARCH PROJECTS AGENCY

Under ARPA Order No. 393, Amendment No. 4

Monitored by the

U. S. ARMY MISSILE COMMAND

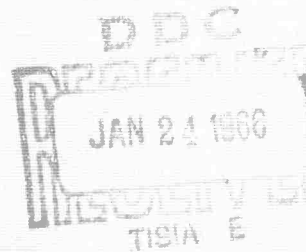
Redstone Arsenal, Alabama

Under Contract No. DA-01-021-AMC-12050(Z)

(Part of Project DEFENDER)

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FORWARD

This report summarizes the status and progress of the re-entry physics research activities at General Dynamics/Convair under Contract DA-01-021-AMC-12050(Z) during the period 1 July 1965 to 30 December 1965.

Although the major portion of this work was supported by ARPA funding through the U. S. Army Missile Command, part of the work related to the construction of a gas handling system for preparing gas mixtures containing ozone was supported by General Dynamics/Convair research funding.

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ABSTRACT

A summary is given in this report of the re-entry physics research conducted at General Dynamics/Convair for the six-month period ending 30 December 1965. Preparations for the study of the rate of the radiative recombination of atomic oxygen and carbon monoxide and of the emission intensity spectral distribution accompanying the recombination to be induced by shock heating gas mixtures containing O_2 , O_3 , CO, CO_2 and Ar are discussed. The topics of discussion include the construction and operation of optical and gas handling systems, the synthesis, stability and decomposition rates of ozone in the gas handling system and preliminary experiments on the emission intensity of recombination. From the latter experiments, conducted at temperatures and pressures near $3000^\circ K$ and 1 atm, a relative intensity spectral distribution curve was derived and found to be shifted to shorter wavelengths with respect to a similar curve derived from flame measurements at about $1500^\circ K$ and comparable pressures. Also, measurements of the induction period preceding formation of measurable quantities of CO_2 in shock heated gas mixtures containing CO, O_2 and Ar were made at temperatures between 1450° and $2260^\circ K$ and pressures as low as 0.01 atm in a 24" internal diameter shock tube.

SHOCK TUBE STUDIES

INTRODUCTION

B. F. Myers

The goal of the present studies on chemical kinetics and the related radiation phenomena is to contribute to the interpretation and quantitative description of observations made during re-entry on the flow around and behind vehicles with ablating materials containing carbon.

During the present reporting period, preparatory work for the measurement of the rate of the radiative recombination of atomic oxygen and carbon monoxide and of the emission intensity spectral distribution accompanying the recombination was completed. The results of preliminary measurements on the emission from the radiative recombination induced by shock heating mixtures containing CO, O₂ and Ar, indicated that the intensity levels were adequate for recording the emission over the wavelength interval 2500Å to 8000Å and at pressures and temperatures of the order of 1 atm and 3000°K. In addition, measurements of the induction period preceding formation of measurable amounts of CO₂ in shock heated gas mixtures containing CO, O₂ and Ar, were made at pressures as low as 0.01 atm and temperatures in the range 1450° to 2260°K in a 24" internal diameter shock tube.

In the forthcoming report period, it is anticipated that measurements of the reaction rate and spectral distribution of emission characteristic of the O and CO recombination will be made by shock heating mixtures containing CO, O₂, C₃, CO₂ and Ar of compositions for which an equilibrium

state will be closely approximated within about 20 μ sec (laboratory time) after passage of the shock front. Thus a near equilibrium state will be rapidly established before the effects of shock wave attenuation or boundary layer growth become important. Investigation of the chemical kinetics governing carbon monoxide oxidation at low pressures will also be continued.

SHOCK TUBE STUDY OF THE RADIATIVE RECOMBINATION OF O AND CO

B. F. Myers, E. R. Bartle and P. R. Erickson

Research activities directed toward the quantitative study of the radiative recombination of O and CO in a high temperature system at equilibrium behind incident shock waves generated in a 3" internal diameter shock tube facility are discussed in the following sections.

Optical System

The construction of the optical system for measurement of the absolute emission intensity accompanying the recombination of O and CO was completed. A schematic diagram of the arrangement of the elements of the system is shown in Figure 1. The entrance slit, S, of the f/12 Hilger Medium Quartz Spectrograph can be imaged either on the inner surface of the shock tube window, S', or on the surface of a tungsten ribbon filament lamp, F, (G.E. 30A/T24/7) which was calibrated by the Eppley Laboratory, Inc. against National Bureau of Standards reference standard lamps. With the swivel-mounted, spherical mirror, M₃, in the arresting position a, S' is the image plane of the spectrograph entrance slit whereas with the mirror in the arresting position b, F is the image plane. The optical paths corresponding to the two arresting positions of the mirror, M₃, contain identical flat surface mirrors, M₁ and M₂, and identical quartz windows to provide the same transmission and reflection losses for the radiation from either the lamp or the shock heated gases; furthermore, radiation from either of the latter sources transmitted to the spectrograph fills the same solid angle. Radiation accepted by the spectrograph is dispersed and passes through a slit system placed at the

image plane of the spectrograph. For initial measurements of the emission intensity of the radiative recombination of O and CO, the center wavelengths of the six slits were approximately 3000Å, 4000Å, 4500Å, 5500Å, 6500Å and 8000Å with bandpasses of 196Å, 195Å, 186Å, 277Å, 82Å and 394Å, respectively. The wavelength calibration of the slits was made by exposing a photographic plate, placed behind the slit system, to a source with a continuous spectrum; the distances of the boundaries and midpoints of the exposed sections from a reference mark were then used to obtain the corresponding wavelengths within an error of less than 3Å from a dispersion curve constructed with the aid of iron and copper spectra. In addition, provision was made for installing a set of six additional slits centered at 2500Å, 3000Å, 3500Å, 5000Å, 6000Å and 7250Å so that intensity measurements may be made at a total of eleven spectral intervals in two experiments each employing five different spectral intervals and one common spectral interval. Flat surface mirrors were placed behind the slits in order to direct the incident radiation onto the photocathode surfaces of six photomultipliers. The Carlson Direct Analyzer attachment was modified to accommodate these photomultipliers and the emitter followers for each. For detection of incident radiation in the spectral interval between 2500Å and 5500Å, four RCA type IP28, 9-stage photomultipliers with S-5 spectral response, two of which have quartz windows, were employed and in the spectral interval between 5500Å and 8000Å, two RCA type 7265, 14-stage photomultipliers with S-20 spectral response were employed.

The response of the detector system to radiation from the tungsten ribbon filament lamp was checked for linearity. The results of this test are shown in Figure 2 where the output signal of the detector system is plotted versus the radiance of the standard lamp. The straight lines

are drawn through the data with unit slope and demonstrate, for a range in the values of the output signal and lamp radiance greater than two orders of magnitude, that a linear relation between these quantities exists for each of the six channels in which the photomultiplier-detectors are positioned to receive radiation over spectral intervals centered approximately at 3000Å, 4000Å, 4500Å, 5500Å, 6500Å and 8000Å. In addition, an initial intensity calibration in terms of the radiance of the standard lamp was made for each of the six channels using the chopper to obtain an AC signal.

Gas Handling System

The construction of the gas handling system for preparing test gas mixtures containing ozone was completed. A diagram of the system is shown in Figure 3. The materials used for construction of that portion of the system in contact with ozone under normal operating conditions were limited to glass, stainless steel (304 or .16), teflon and H_2SO_4 , the manometric fluid; these materials do not readily react with ozone or promote its catalytic decomposition at ambient temperatures. Also, before ozone was handled in the system, all parts were thoroughly cleaned with trichloroethylene, methyl alcohol and distilled water in that sequence to remove the remaining traces of machining oil and of other organic contaminants.

The system includes a 15% mixing tank for preparing mixtures containing O_2 , CO, CO_2 and Ar and a 10% mixing tank for preparing mixtures of O_3 with gas mixtures from the 15% mixing tank. In order to promote rapid preparation of homogeneous gas mixtures and to minimize the effects of possible slow reactions of ozone with the other gas mixture constituents, opposing paddles which can be driven magnetically by an external motor are incorporated in both tanks. The partial pressures of the gas mixture

constituents are measured during preparation of the mixtures with reservoir-type, H_2SO_4 manometers or a K_2SO_4 buffered-Hg manometer which have precision bore, 1/4" I.D., glass columns connected to stainless steel reservoirs. Between each reservoir and the remainder of the gas handling system is a "dry ice" trap into which is inserted a continuous spiral¹ composed of a layer of 0.003" thick stainless steel corrugated to a depth of 0.039" and a second layer of plane 0.003" thick stainless steel in order to provide an effective trap for sulfuric acid vapors.

Ozone is synthesized in a static ozone generator² which is surrounded by liquid nitrogen during ozonization. The starting material for the synthesis is electrolytic laboratory grade O_2 (Liquid Carbonic) containing less than 10 ppm of impurities due to H_2 , H_2O and N_2 . The rate of ozonization attained under the present operating conditions is 1.54 gms O_3 /atm-hr, where the pressure is in reference to the oxygen pressure in the ozonizer. With the static ozone generator, complete conversion of the initial charge of oxygen to ozone can be obtained, if desired. However, under normal operating conditions, about 95% conversion of O_2 to O_3 is obtained and the excess O_2 is then pumped off. Excess ozone is removed from the gas handling system by passage through a heat trap composed of a 2"x28" tube filled with 0.005" spirally-wound copper held at 200°C, this treatment promotes the catalytic decomposition of ozone and thus retards the accumulation of ozone in the cold trap which isolates the roughing pump from the main system.

The ultimate minimum pressure attained and the corresponding leak rate measured in that part of the gas handling system consisting of the 154

1. D. Alpert, Rev. Sci. Inst. 24, 1004 (1953).
2. B. F. Myers, E. R. Bartle and P. R. Erickson, unpublished results.

mixing tank and either the H_2SO_4 or H_2SO_4 buffered-Hg manometer were 1.5×10^{-4} mm Hg and at this pressure, 2.0×10^{-4} mm Hg/min, respectively, as measured with an ionization gauge attached directly to the mixing tank; with only the 15l mixing tank being evacuated, the pressure attained was 3×10^{-5} mm Hg with a corresponding leak rate of 2×10^{-5} mm Hg/min at a pressure of 3×10^{-5} mm Hg. With that part of the gas handling system consisting of the 10l and 15l mixing tanks plus the H_2SO_4 and H_2SO_4 buffered-Hg manometers, the minimum pressure attained was 1.7×10^{-4} mm Hg and the leak rate was determined to be 2.2×10^{-4} mm Hg/min at 1.7×10^{-4} mm Hg. Under normal operating conditions, these leak rates result in an increase in the contamination level of test gas mixtures prepared in the 15l and 10l mixing tanks by less than 1 and 11 ppm, respectively. In that part of the system consisting of the ozonizer and the "dry ice" trap which isolates the oxygen cylinder from the ozonizer, the minimum pressure attained upon evacuation was 3×10^{-5} mm Hg and a leak rate of 1.3×10^{-4} mm Hg/min at 3×10^{-5} mm Hg was subsequently measured. This leak rate results in an increase of the contamination level of the oxygen charge to the ozonizer of less than 2 ppm under the normal operating procedure.

The Analysis and Stability of Ozone

Three methods were employed for the quantitative analysis of ozone. For pure ozone and for gas mixtures with a known mole fraction of ozone, the absolute quantity of ozone was determined from a knowledge of the total gas pressure of a sample in a pyrex flask whose volume was known to within an error of 0.3%. The quantitative analysis of ozone by this pressure-volume measurement was compared with the analysis made by a

standard³ titration procedure in which the ozone is absorbed in a buffered KI solution and subsequently titrated with a standardized $\text{Na}_2\text{S}_2\text{O}_3$ solution. To collect the ozone with the buffered KI solution, the flask containing the ozone sample was connected through a ground glass joint to a second flask containing the absorbing solution in an atmosphere of argon. After opening the stopcock of the first flask, the assembly was shaken for several minutes. In using the procedure, it was found that the pressure-volume measurement and the titration method agreed to within an error of $\pm 2\%$. A second procedure for collecting the ozone by flushing the ozone with argon through a gas washing bottle containing the buffered KI solution was found to result, with the present apparatus, in a recovery of less than the quantity of ozone expected on the basis of pressure-volume measurements. A third analytical method for determining the quantity of ozone in mixtures of ozone and argon was devised by adding a gas sample directly to the charcoal column of a Perkin Elmer Model 154D Vapor Fractometer. The ozone readily decomposed in the presence of the column material to form oxygen; from the chromatographic analysis of the quantity of oxygen eluted, the original ozone concentration of the gas mixture sample was computed. The quantity of ozone determined chromatographically agreed with that quantity expected on the basis of pressure-volume measurements to within an error of $\pm 1.5\%$. Additional preliminary experiments on the chromatographic analysis of mixtures of O_3 with CO , O_2 , CO_2 and Ar indicate that in the presence of the column material and under the present operating conditions ozone decomposes to form O_2 and, by reaction with CO , to form CO_2 where the quantity of CO_2 formed is proportional to the quantity

3. D. H. Byers and B. E. Saltzman, "Ozone Chemistry and Technology," Advances in Chemistry Series No. 21, American Chemical Society, Washington, D.C., 1959, p. 93.

of CO originally present in the gas mixture sample. From an analysis of the increases in the concentrations of O_2 and CO_2 eluted and of the decrease in the concentration of eluted CO, the original ozone concentration of the gas mixture sample was determined; the computed ozone concentrations agreed with the concentrations determined by pressure-volume measurements to within an error of $\pm 8\%$ for gas mixtures containing about 1.7 mole % ozone.

The stability of ozone and of gas mixtures containing ozone during residence in the 10% stainless steel mixing tank was determined by monitoring the ozone concentration as a function of time. With pure ozone and a mixture of ozone in argon containing 2 mole % ozone, the rates of decomposition were 0.36 mole % O_3 /hr over a period of 19 hrs and 0.135 mole % O_3 /hr over a period of 100 hrs, respectively. The contribution of reactions of impurities with ozone or of the surface catalyzed decomposition of ozone to these rates is small since the rates expected on the basis of the homogeneous gas phase decomposition of ozone⁴ under the present experimental conditions are approximately 0.33 mole % O_3 /hr for pure ozone and 0.10 mole % O_3 /hr for a dilute ozone-argon mixture. When the ozone was first added to the 10% mixing tank, a very rapid and extensive decomposition of ozone occurred in spite of the thorough cleaning procedure used to prepare the gas handling system; however, on subsequent additions of ozone to the mixing tanks, only the decomposition rates given above were recorded.

When mixtures containing ozone and carbon monoxide as well as oxygen, carbon dioxide and argon were prepared in the 10% stainless steel mixing tank, an initial, rapid decomposition of ozone occurred which was shown to result from impurities in the carbon monoxide and not to occur when

4. S. W. Benson and A. E. Axworthy, J. Chem. Phys. 26, 1718 (1957); W. M. Jones and N. Davidson, J. Amer. Chem. Soc., 84, 2868 (1962).

only oxygen or carbon dioxide in addition to argon was mixed with ozone. Following this initial decline in ozone concentration, an average rate of decomposition of about 0.24 mole % O_3 /hr was observed for several gas mixtures with nominal compositions of 2.5% O_3 + 9.7% O_2 + 19.5% CO + 4.9% CO_2 + 63.4% Ar; the expected rate of the homogeneous gas phase decomposition of ozone⁴ in these mixtures is approximately 0.14 mole % O_3 /hr. The possible contribution to this difference in the observed and calculated rates of ozone decomposition by additional, slowly reacting impurities in the CO will be assessed after installation of a liquid nitrogen cold trap in the CO supply line of the gas handling system to permit purification of the CO by fractional vaporization.

Preliminary Experiments on O-CO Recombination

Possible contributions, from transitions other than those involved in the radiative recombination of atomic oxygen and carbon monoxide, to the total radiation emitted at equilibrium by a gas sample containing carbon, oxygen, argon and impurities present in the components of the gas mixture were investigated by shock heating test gases containing Ar and mixtures of Ar with CO, O_2 and CO_2 . At temperatures of approximately 3000°K and pressures in the range between 1.0 and 1.3 atm, shock heating pure argon and mixtures of argon with 10-20 mole % O_2 or 15 mole % CO_2 resulted in no detectable radiation in the six spectral channels (see section 1). In contrast to these observations, shock heating a mixture of 20 mole % CO in argon to approximately 3000°K and 1 atm resulted in a measurable oscilloscope signal at the spectral interval centered at 3000Å but in no detectable signals at the other spectral intervals. The source of the radiation at about 3000Å has not yet been identified.

To ascertain the emission intensity accompanying the radiative recombination of O and CO, three preliminary experiments were conducted by shock heating mixtures with the nominal compositions of 10% O₂ + 20% CO + 5% CO₂ + 65% Ar in two cases and of 10% O₂ + 20% CO + 70% Ar in the third case to temperatures and pressures at equilibrium of 3029°K and 1.10 atm, 3049°K and 1.05 atm, and 3170°K and 1.11 atm, respectively. In all experiments, substantial oscilloscope signals were recorded in each of the six spectral channels. To reduce the oscilloscope signals to values of relative intensity, each signal voltage was normalized with respect to the intensity calibration factor for the spectral channel in which it was recorded and the product of the concentrations of O and CO at equilibrium; the relative intensity values were derived with the aid of a smooth curve drawn through the normalized data when plotted as a function of wavelength. The relative intensity spectral distribution is shown in Figure 4 where the average value of the computed relative intensity at a given wavelength for the three experiments is plotted. The flags indicate the range of the measured values. It is evident from Figure 4 that the relative intensity decreases monotonically from a maximum value at about 3400Å to a near zero level at about 7500Å when the smooth curve through the data points is extrapolated beyond the datum at 6500Å. Furthermore, as shown in Figure 4, a comparison of this relative intensity spectral distribution curve with published data⁵ obtained at 1520-1800°K and 0.16 to 1.0 atm in a CO + H₂ + air flame over the wavelength interval 3500Å to 6000Å demonstrates that the entire relative intensity spectral distribution curve is shifted to smaller wavelengths at the higher temperatures of the present experiments,

5. W. E. Kaskan, Comb. & Flame 3, 39 (1959).

i.e., about 3000°K . In contrast to the near zero level of relative intensity expected at wavelengths greater than about 7500\AA , it was found in these three preliminary experiments that in the spectral interval centered at 8000\AA , a signal was obtained which corresponded to a relative intensity of 0.536 ± 0.039 . While the source of this radiation has not yet been determined, the radiation was not detected in experiments by shock heating Ar or Ar and O_2 , or CO, or CO_2 .

The reliability of the relative intensity data presented in Figure 4 is adversely affected by three factors, at least. The oscilloscope signals were measured, somewhat arbitrarily, at approximately $160\text{ }\mu\text{sec}$ (laboratory time) after passage of the shock front beyond the measuring station in a region of the signal-time profile where the signal was sensibly constant. It was thus assumed that the gas sample had closely approached the equilibrium state at a distance behind the shock front corresponding to the time quoted above. The constancy of the oscilloscope signal was the only indication obtainable from the experimental results that an equilibrium state was attained. Two other factors which will affect the reliability of the data are shock wave attenuation and boundary layer growth; both of these will become more important as the distance at which measurement is made behind the shock front increases. The bulk of the experiments to be performed in measuring the radiative recombination of O and CO, as outlined in the introduction are designed to avoid the adverse factors affecting the reliability of the preliminary experiments which have been described.

SHOCK TUBE STUDY OF CO_2 FORMATION AT LOW DENSITY

B. F. Myers, E. R. Bartle and P. R. Erickson

The study of the formation of CO_2 at high temperatures and low densities in shock heated gas mixtures containing CO and O_2 was started in the General Dynamics/Convair 24" diameter shock tube facility.⁶

Preliminary test runs in pure CO_2 indicated that the shock transit time across the observed gas slab in the optical system of the 24" diameter shock tube can be neglected by comparison with the induction time preceding the onset of measurable CO_2 formation expected at low density.

Measurements of the formation of CO_2 behind incident shock waves traveling through mixtures with the nominal composition of 10% O_2 + 20% CO + 70% Ar were made at pressures between 0.36 and 0.55 atm and at temperatures between 1450° and 1920°K in order to compare the operation of the 24" diameter shock tube facility with that of the 3" diameter shock tube facility under similar conditions of pressure, temperature and gas mixture composition. The measured induction times preceding the onset of rapid electronic ground state CO_2 formation were converted to an induction period parameter^{7,8} (i.e., the induction time at one atmosphere pressure measured in particle time); in Figure 5, the induction period parameters, $\alpha_1/\text{atm } \mu\text{sec}$, are plotted versus the reciprocal absolute temperature for the induction times measured in both the 3" and 24" diameter shock tubes. It is evident that within the scatter of the experimental data, the induction period parameters derived from the 3" and 24"

6. E. R. Bartle and K. G. P. Sulzmann, General Dynamics/Convair Report GDC-DBE65-011, June, 1965.
7. K. G. P. Sulzmann, B. F. Myers and E. R. Bartle, J. Chem. Phys. 42, 3969 (1965).
8. B. F. Myers, K. G. P. Sulzmann and E. R. Bartle, *ibid*, 43, 1220 (1965).

diameter shock tube experiments are identical.

Further experiments were then conducted in the 24" diameter shock tube with test gas mixtures with the nominal composition of 10% O_2 + 20% CO + 70% Ar at lower pressures between 0.08 and 0.10 atm and at temperatures between 1450° and 2260°K. Again it is clear from Figure 5 that within the scatter of the experimental data, the measured induction period parameters were identical to those previously obtained with both the 3" and 24" diameter shock tubes. It may be concluded from these experiments that for mixtures with the composition of 10% O_2 + 20% CO + 70% Ar, the induction time, τ_i , (measured in particle time), preceding the onset of rapid CO_2 formation scales linearly with the total gas pressure for temperatures between 1450° and 2260°K and pressures between 0.08 and 1.55 atm, a 20-fold range in pressure. Additional experiments have also been conducted at pressures of 0.01 to 0.05 atm and temperatures between 1700° and 3300°K but the results have not yet been analyzed.

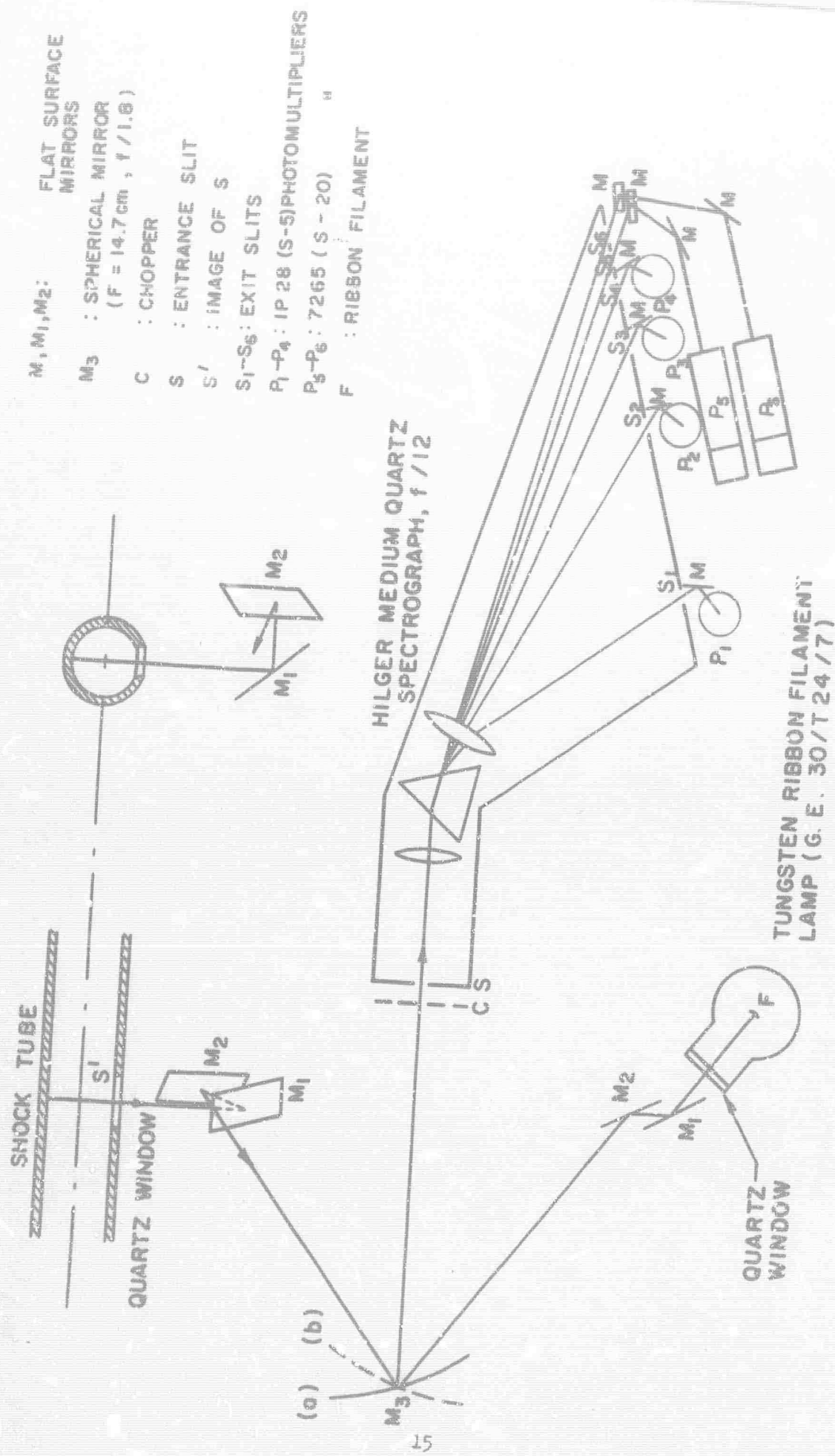


FIGURE 1 : OPTICAL ARRANGEMENT OF ULTRAVIOLET - VISIBLE DETECTION SYSTEM

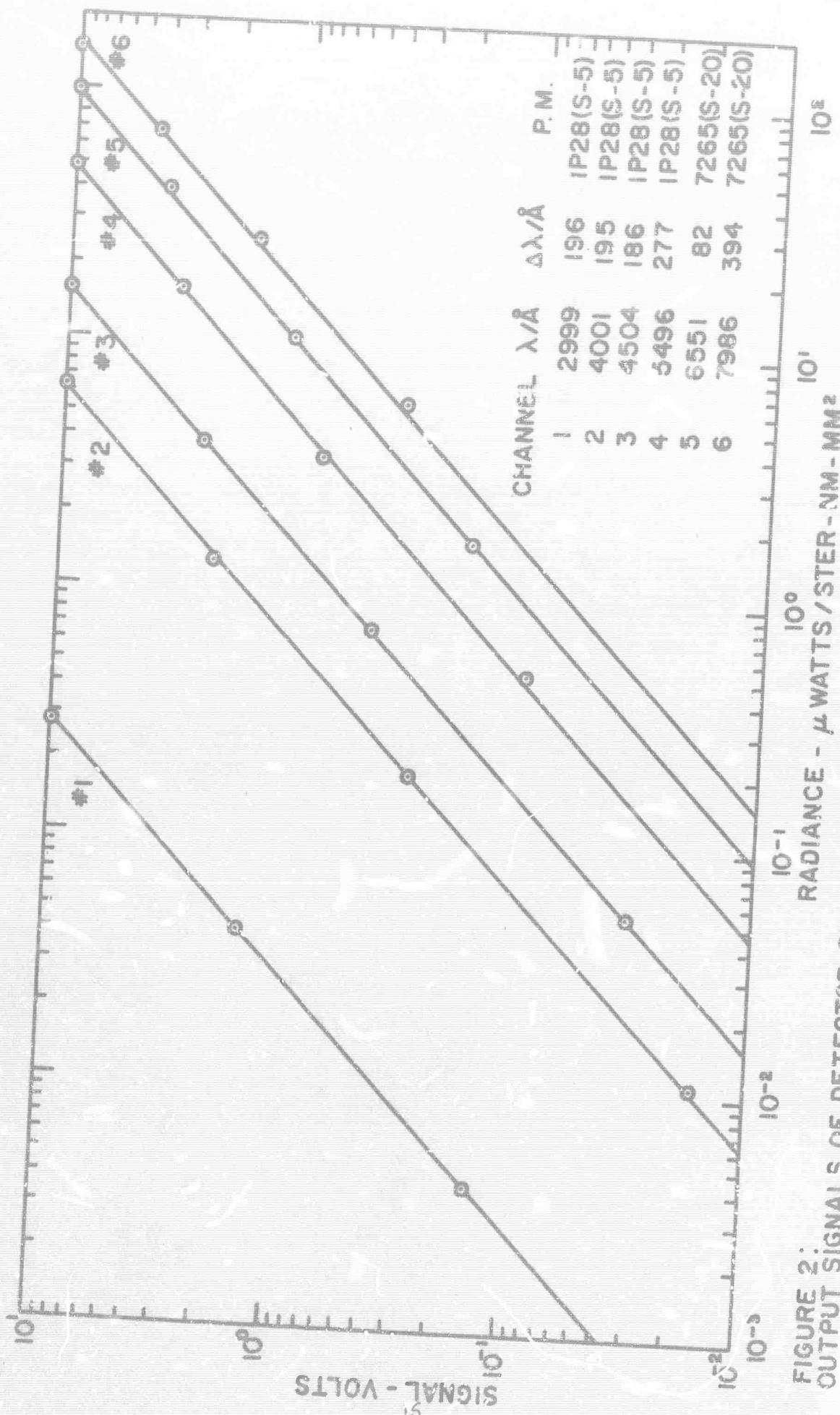


FIGURE 2:
 OUTPUT SIGNALS OF DETECTOR SYSTEM SPECTRAL CHANNELS vs RADIANCE OF A STANDARDIZED
 TUNGSTEN RIBBON FILAMENT LAMP. THE LEGEND GIVES THE CHANNEL CENTER WAVELENGTH
 AND BAND PASS IN \AA AND THE TYPE OF PHOTOMULTIPLIER (P.M.) WITH THE CATHODE SPECTRAL
 RESPONSE DESIGNATION. LINES THROUGH DATA ARE DRAWN WITH UNIT SLOPE.

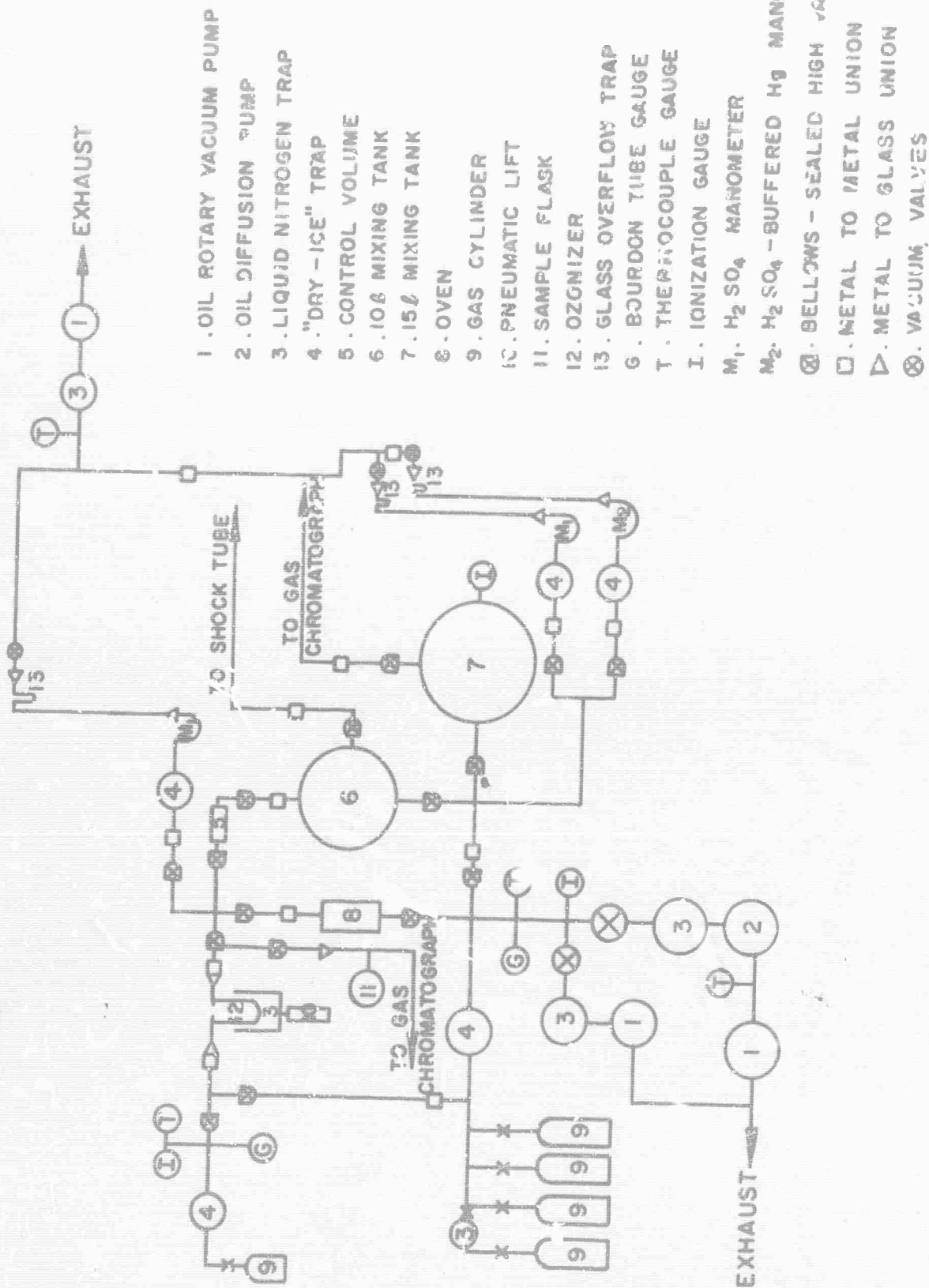


FIGURE 3 : GAS HANDLING SYSTEM

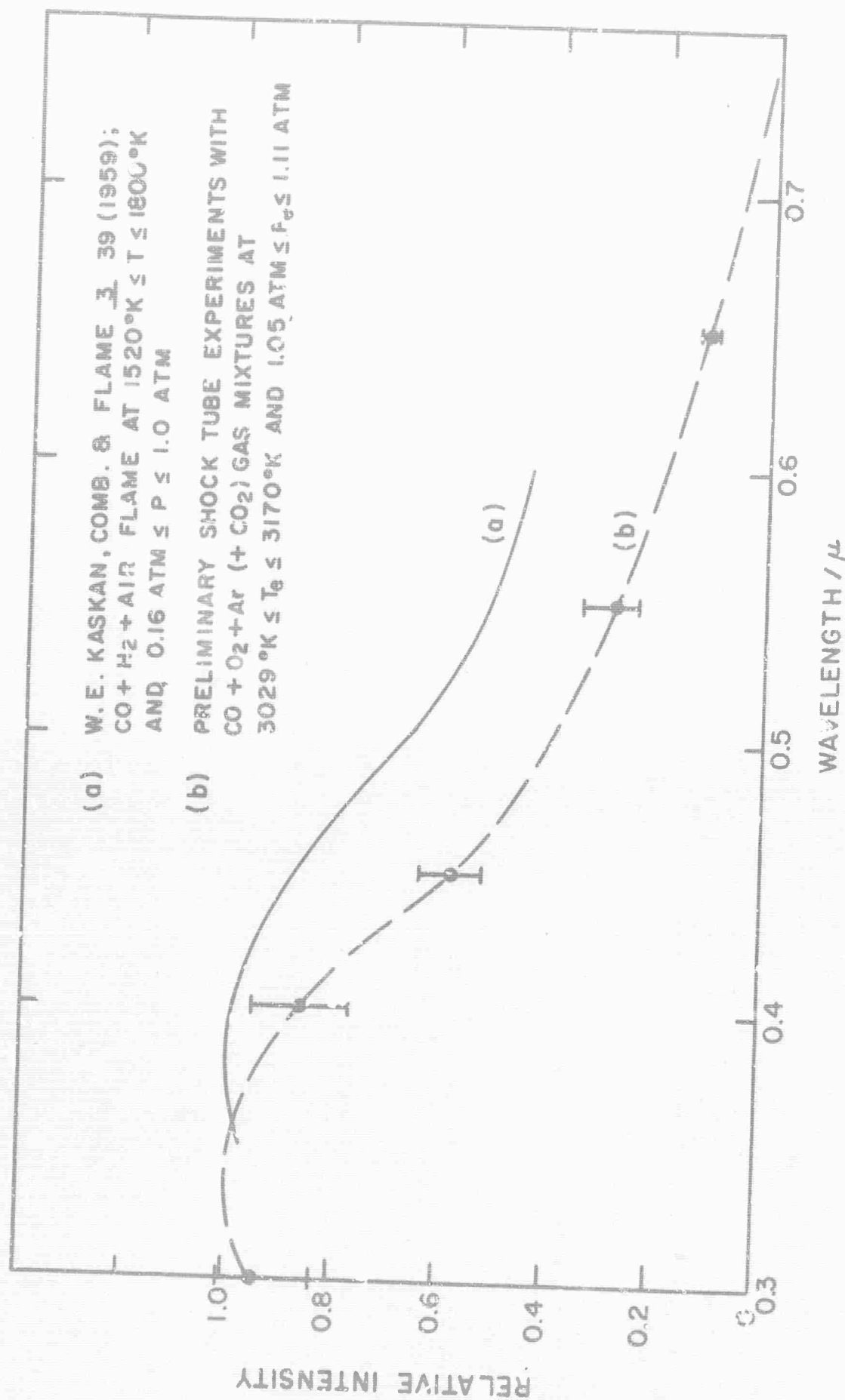


FIGURE 4 : RELATIVE INTENSITY SPECTRAL DISTRIBUTION FOR THE "CO-FLAME BANDS".
THE SOLID CIRCLES ARE THE AVERAGE OF THREE EXPERIMENTS AND THE
FLAGS INDICATE THE RANGE OF DERIVED VALUES

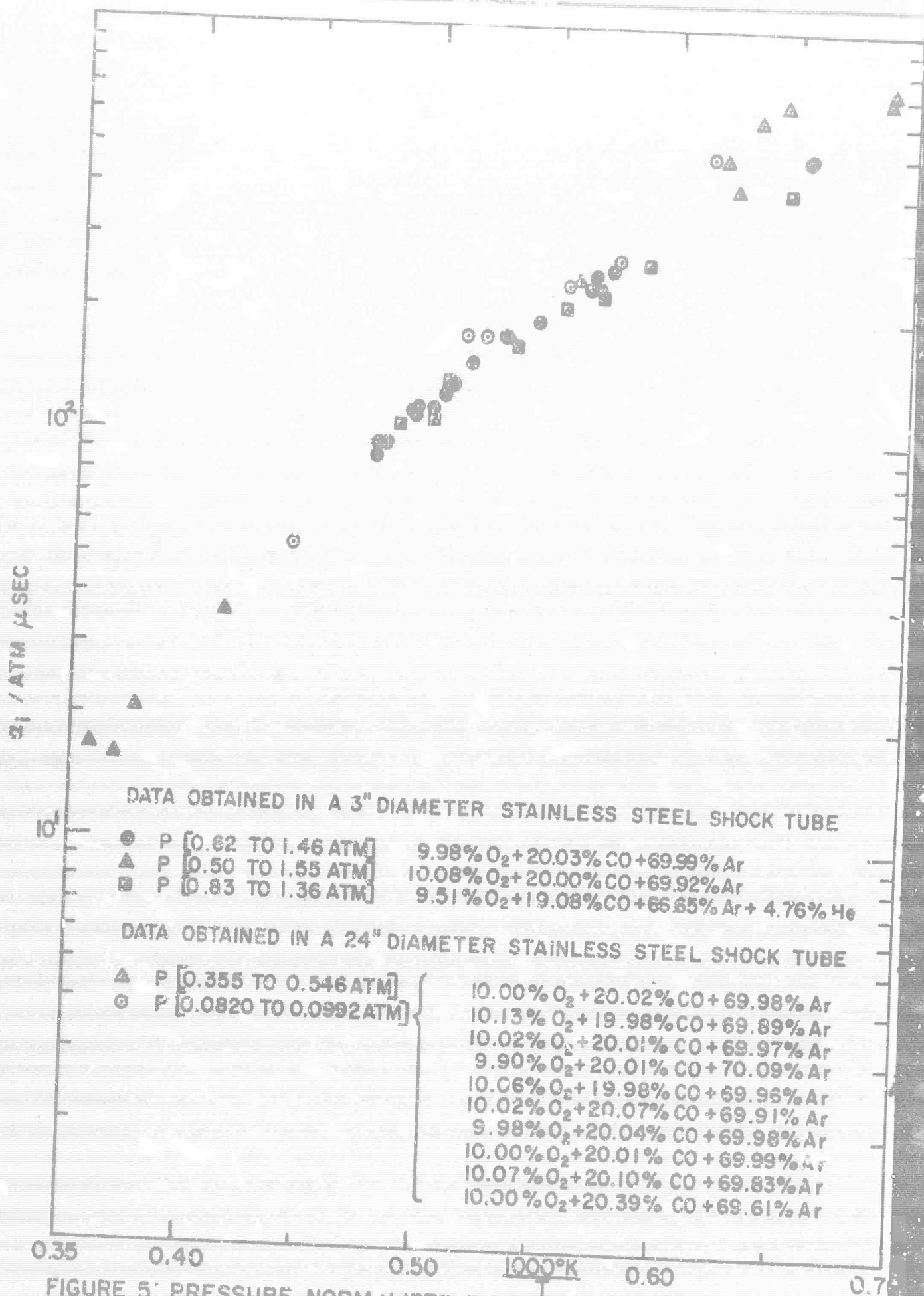


FIGURE 5: PRESSURE NORMALIZED INDUCTION PERIOD, α_i , vs THE DIMENSIONLESS PARAMETER, $1000^\circ\text{K} / T$

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